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## MICROSTRUCTURAL CHARACTERIZATION OF THE HRSI THERMAL PROTECTION SYSTEM FOR SPACE SHUTTLE

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# MICROSTRUCTURAL CHARACTERIZATION OF THE HRSI THERMAL PROTECTION SYSTEM FOR SPACE SHUTTLE

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## SUMMARY

Components of the space shuttle high temperature reusable surface insulation (HRSI) system have been microscopically characterized, both separately and as a system, to obtain information needed for stress analysis models of the thermal protection system. A tension specimen of the HRSI system was loaded in steps and was microscopically observed at each load condition to demonstrate the tension failure mode associated with strain isolation pad (SIP) behavior. A local failure occurred which could be associated with transfer of load through transverse fibers in the SIP.

Stress concentrations attributed to the SIP behavior have necessitated strengthening of the HRSI by densification of the RSI at the bondline. An HRSI tile has been microscopically characterized after the densification process. The densified surface layer blended into the RSI which caused a gradual change in density. The gradation in density does not appear to represent a sharp discontinuity in elastic modulus between the densified layer and the parent material.

## INTRODUCTION

The primary insulation used to protect up to 70 percent of the space shuttle orbiter's exterior surface on reentry is an externally attached, rigidized fibrous silica, designated LI-900 or LI-2200, which has been machined into tiles approximately 15 to 20 cm (6 to 8 in.) square. These tiles, together with a strain isolation material, make up the High or Low Temperature Reusable Surface Insulation (HRSI or LRSI) systems which are described in detail in reference 1. The tiles are bonded to the strain isolation pad (SIP) which in turn is bonded to the orbiter aluminum alloy skin structure using a silicone based room temperature vulcanizing

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(RTV-560) adhesive (fig. 1). The SIP is required to isolate the tiles from strains due to thermal expansion of the orbiter structure and skin deflections due to aerodynamic loads. The SIP is a felted mat of Nomex fibers. The majority of these fibers are oriented in the plane of the mat. Others are normal (transverse) to plane of the mat. The transverse fibers hold the mat together and transfer loads from the tile to the skin. Tension failures of HRSI test tiles at unexpectedly low stress levels have been attributed to the stress concentrations in the RSI at these transverse fibers. To redistribute the stresses in the RSI, a tile densification procedure has been implemented in which the density near the bondline is increased to increase local strength.

This paper reports results of microscopic characterization of the LI-900 HRSI system components and densified LI-900 RSI. Studies of the system were needed to better understand the mechanisms involved in the transfer of tensile loads through the SIP and to obtain information pertinent to the modeling of the SIP in stress analyses. Densified LI-900 RSI was studied to insure that gradation of densifying material was smooth enough so that sudden changes in elastic modulus were not introduced. Also of interest was the effect of densification on the ability of densified tiles to vent air pressure differentials.

## MATERIALS

### RSI and Densified RSI

Specimens of LI-900 RSI used in the investigation were taken from the interior of a tile having Lockheed serial number VT70-090104-014-008168. The tile had a class 2 coating (see fig. 1) and was waterproofed. Specimens of densified LI-900 RSI were taken from a tile having Lockheed serial number V070-395904-006-008030. This tile, also with class 2 coating, was obtained from Kennedy Space Center in the densified condition. Neither tile had been subjected to previous testing.

## SIP

SIP specimens used in this investigation were taken from a 305 mm (12 in.) square by 4.0 mm (0.16 in) thick piece of SIP with RTV-560 transfer coat on one side. The transfer coat was protected by a peel ply. The SIP was supplied to NASA by Rockwell International, Space Systems Division.

## ADHESIVES

RTV-560, a room temperature vulcanizing silicone based adhesive, was used for all bonding.

## PROCEDURE

### Preparation of Specimens

RSI Specimens. - Specimens for routine scanning electron microscopy were cut from the approximate geometric center of the RSI tile. The specimens were mounted for a viewing plane parallel to the tile face and a plane perpendicular to the face.

SIP Specimens. - Specimens for routine scanning electron microscopy were cut from the supplied SIP. They were mounted for viewing the cross-section and the uncoated face.

Densified RSI Specimens. - Three specimens were prepared from the densified tile for examination in the scanning electron microscope (SEM). The specimens were cut from the tile at locations shown in figure 2. They were then placed in a furnace at 675 K (750 °F) for 12 to 15 hours to remove any volatiles introduced by the densification process (this procedure seemed to help in maintaining the conductive coating on the specimens in the SEM vacuum). The specimens were mounted for viewing the tile cross-section.

SIP/RSI Tension Specimen. - The SIP/RSI specimen was fabricated as follows: A 6mm (0.23 in) thick coupon of RSI machined from tile number VT70-090104-014-8168 was bonded to the coated side of a 25 mm (1 in) square coupon of SIP. The thickness direction of the RSI coupon corresponded to the

thickness direction of the parent tile. The joined SIP/RSI was then bonded between two 3 mm (0.125 in) thick by 25 mm (1 in) square aluminum plates. The plates were carefully aligned with the SIP coupon. The RSI coupon was intentionally cut oversize in the flatwise dimension to maintain flatness and good bonding to the edge of the transfer coat on the SIP coupon (handling of RSI tends to round off edges). Standard tile bonding procedures were used for the SIP/RSI joint (Rockwell Int. MPP No. 106 M319 M03 dated 5/10/79). During curing of the adhesive, the specimen was set up with the RSI toward the bottom so that any excess adhesive would flow away from the edge of the SIP transfer coat. After curing, the excess RSI and adhesive were carefully trimmed from each side. A cross-sectional view of the specimen is shown in figure 3.

#### Tension Loading of TPS Specimen

Tension loads were applied to the TPS specimen as follows. A hole was drilled through the center of one aluminum plate and through the TPS. The hole was threaded so that a screw could be advanced against the opposite plate to load the specimen. Tension loads applied to the specimen were estimated from deflections of the SIP using secant modulus values for SIP of 86.2 kPa (12.5 psi) at a 13.8 kPa (2 psi) stress and 183 kPa (26.5 psi) at both 48.3 kPa (7 psi) and 110.3 kPa (16 psi) stress levels. The RSI and the RTV bond were assumed to be very rigid compared to the SIP. The specimen was examined in the SEM before load was applied and under each of the static loads previously noted.

#### Microscopy Techniques

All specimens were examined using standard scanning electron microscopy techniques for non-conductive materials. The specimens were coated with a vapor deposited gold-palladium alloy to produce the electrically conductive surfaces required for good SEM results. Careful cleaning using a gentle vacuum source before coating also improved results. However, cleaning

procedures could not be used on SIP specimens without causing increased fraying of the cross-sections.

## DISCUSSION AND RESULTS

### SIP/Transfer Coat

The SIP was examined in detail in the SEM to gain an understanding of the relationship between its microstructure and its behavior under transverse loading. Examples of SIP microstructure are given in figure 4. The in-plane views are of the uncoated face of the SIP and the cross-section view shows the SIP/RSI transfer-coat interface. Several structural features of the SIP are apparent in these photomicrographs. At low magnification (fig. 4a), a pattern of dimples can be seen on the uncoated face of the SIP. A higher magnification view of one of these dimples (fig. 4b) clearly shows that the dimples are locations of transverse fiber bundles. These bundles are spaced about 1.5 mm (0.06 in) apart. The cross-section view in figure 4c represents a linear distance of about 3 mm (0.12 in). The transverse bundle shown is solidly anchored in the RTV transfer coat while the fibers to either side are mostly oriented in the plane of the SIP. Note that the transverse fiber bundle is not perpendicular to the plane of the SIP. This is characteristic of the material. In all cross-sections observed, the transverse fibers are predominantly curved and slanted. These directional characteristics appear to be related to the manufacturing process in a manner analogous to preferred grain orientation in the roll direction of a metal sheet. Thus, the SIP may have longitudinal and/or long transverse characteristics which will influence its short transverse (through-the-thickness) behavior. Another important structural characteristic of the material is that transverse fiber bundles are not always solidly anchored in the transfer coat nor are they always anchored on the SIP/skin bond at the opposite ends. Evidently, anchored fibers are mechanically locked and not chemically bonded to the RTV (see, for instance, locations marked A in figure 4c) which means that fiber pull out could occur under transverse loading.

Some features observed on failure surfaces of tile/SIP tension test specimens appear to result from influences of the SIP structure. Macro-examination of a typical failure surface, the RSI/SIP interface, reveals a random pattern of dimples of varying depths which were not originally present on the surface of the transfer coat. The dimples can readily be explained in terms of the transverse fiber bundles through which tensile loads must be carried (see fig. 5). A stress concentration is produced at the anchor point of the transverse bundle which causes local overloading of the RSI and initiation of failure. Material surrounding the stress concentration point is able to continue elongating so that a dimple is produced in the transfer coat. Upon complete separation and return to the no-load condition, the differential residual strain across the region allows the dimple to remain. Randomness of the dimple pattern and dimple depth can be attributed to variations in load capacity of the transverse bundles associated with the degree of anchoring, fiber breakage, and/or fiber pull out.

#### SIP/RSI Tension Specimen

The SIP/RSI tension specimen shown in figure 3 was observed in the SEM at different loads from zero to failure to determine if individual transverse fiber bundles could be related to initiation of failure. The cross section shown in figure 3 was taken parallel to what is believed to be the "longitudinal" direction of the SIP. SEM photographs taken at the different load conditions are shown in figure 6a. The transverse fiber bundles are slanted and curved in a preferred direction in the no load condition. The transverse fibers or fiber bundles of interest are tagged with arrows A and B so that they can be tracked at different loads. The initial point of separation at failure in the RSI is also indicated by an arrow. The identified transverse elements are more easily seen in the higher magnification SEM photographs of figures 6b and 6c and can be identified as active load-carrying elements because of their behavior with increasing load. At no load in figure 6b, the bundle marked A is preferentially slanted and curved. At a transverse load of about 13.8 kPa (2 psi), the bundle has straightened and



now appears to be slanted in the opposite direction. If the specimen imposed no shear constraints or if the anchor points at each end of the bundle were aligned with the transverse load, the bundle would become perpendicular. The jack screw used to load the specimen constrains shear motions of the RSI relative to the SIP. Therefore, the reversal of the slant direction is indicative of an offset of anchor points at opposite ends of the bundles. This trend throughout the SIP would lead to a coupling of the in-plane deflections with transverse deflections. High magnification SEM photographs of the SIP/transfer coat interface in figure 6c show evidence of poor bonding of fibers to the RTV. The fiber marked B is slanted in the preferred direction at the no-load condition. As the load is increased to about 48 kPa (7 psi) and then to 110 kPa (16 psi), the fiber rotates to a more perpendicular position and increasingly separates from the RTV. The proximity of the transverse elements A and B to the failure initiation point supports the hypothesis that transverse fibers produce a local stress concentration in the RSI which initiates failure. Photoelastic analysis (unpublished work by Paul Cooper, Langley Research Center and R. Prabhakaran, Old Dominion University) has provided additional verification that the transverse fiber bundles produce stress concentrations.

#### Densified RSI

The densification of LI-900 RSI bonding surface is accomplished by infiltration of the surface with colloidal silica mixed with a slurry of larger silica particles. A representative section of the densified tile is shown in figure 7. The densification process forms a nearly continuous dense crust on the bonding surface about 0.5 mm (0.02 in) thick. The density decreases gradually toward the interior of the tile with most of the infiltrated material remaining within about 4 mm (0.16 in) of the bonding surface. Clearly, because of the reduced porosity of the RSI, air venting capabilities of the densified RSI are significantly reduced to depths of at least 2.5 mm (0.10 in) from the densified surface. Figure 8 shows higher magnification SEM photographs of the densified tile section at 2 mm (0.08 in) and 20 mm

(0.8 in) depth. A SEM photograph of undensified RSI is included for comparison. The larger particles in the densifying material are clearly visible at the 2 mm depth. Some colloidal material is seen at the 20 mm depth. However, this is the exception rather than the rule.

## CONCLUSIONS

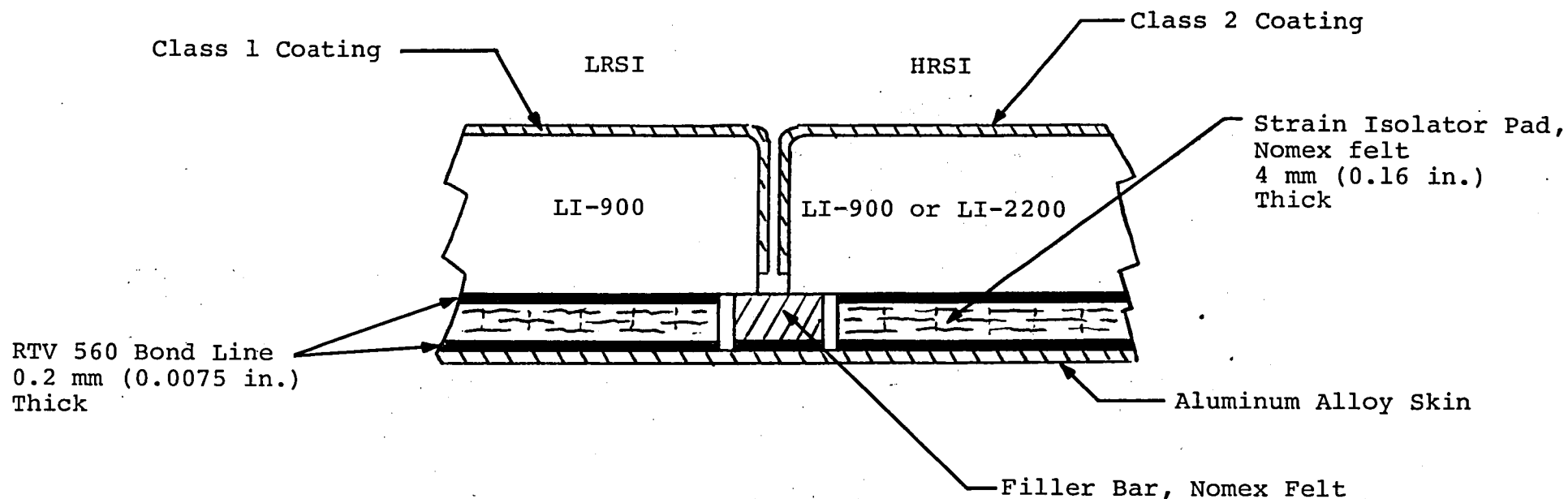
The space shuttle HRSI TPS has been microscopically characterized to better understand the mechanisms involved in transfer of tensile loads through the SIP and to provide information for stress modeling of the SIP. Also, densified LI-900 RSI has been examined to determine if densification is such that abrupt changes in elastic modulus might exist. The following conclusions resulted from this investigation.

1. The structure of the SIP is such that tensile loads must be transmitted to the RSI tiles at discrete points.
2. Local failure due to stress concentration at the SIP/RSI bondline can be directly related to individual transverse fiber bundles.
3. Transverse fiber bundles are not always anchored in the RTV layers at both surfaces of the SIP.
4. Anchored transverse fiber bundles appear to be mechanically locked in the RTV layers rather than chemically bonded.
5. Transverse fiber bundles are not perpendicular to the plane of the SIP but rather they are slanted in a preferred direction and have some curvature. This slanting of the transverse fibers should result in a coupling of deflections in the in-plane and transverse directions of the SIP.
6. Densification of LI-900 RSI produces a dense crust on the surface with a gradual decrease in density toward the interior of the tile. Most of the densifying material (colloidal silica and silica slip) is within 4 mm (0.16 in) of the surface of the densified tile. Traces of colloidal silica have been observed to depths of 20 mm (0.79 in).
7. Because of reduced porosity, the ability of the densified RSI to vent air pressure differentials is expected to be significantly reduced to depths of at least 2.5 mm (0.10 in) below the densified surface.

#### REFERENCE

1. Forsberg, Kevin: Description of the Manufacturing Challenges in Producing the High-Temperature Reusable Surface Insulation for the Thermal Protection System of Space Shuttle. Presented at XIV Congress International Aeronautique, Paris, France, June 1979.





LRSI - Class 1 Coating - Low Temperature Tile (673 K to 923 K - (750°F to 1200°F))  
 White Coating (Emittance = 0.65 at 923 K - (1200°F))

HRSI - Class 2 Coating - High Temperature Tile (923 K to 1533 K - (1200°F to 2300°F))  
 Black Coating (Emittance = 0.8 at 1533 K - (2300°F))

Figure 1.- Space Shuttle Thermal Protection System cross section (from reference 1).

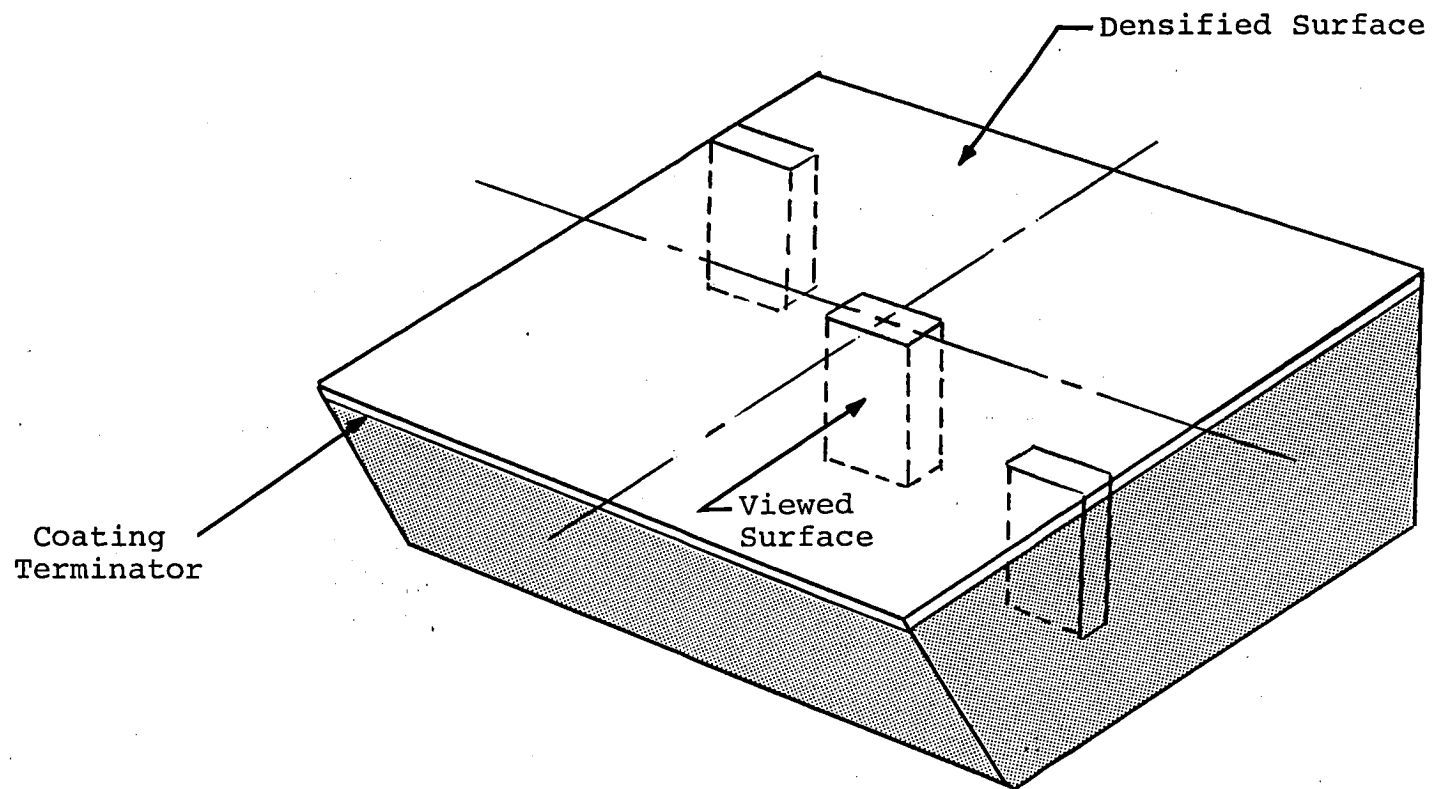


Figure 2.- Locations of SEM specimens taken from densified  
RSI tile number V070-395904-006-008030.

RSI

SIP

Aluminum

RTV

RTV

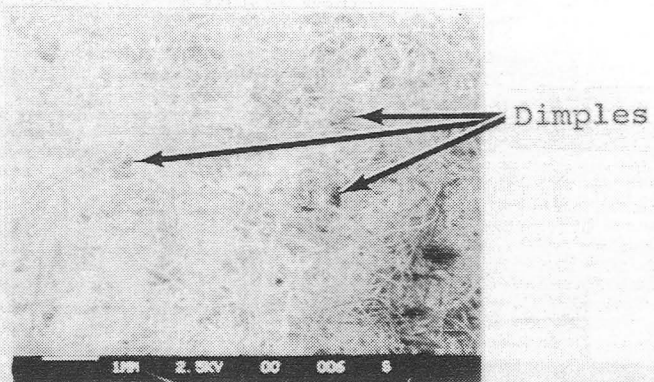
RTV

Aluminum

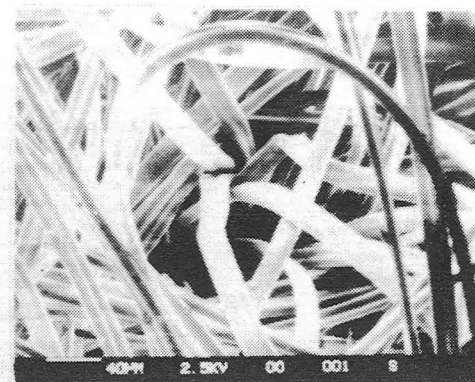
13 mm

Figure 3.- SIP/RSI tension specimen for scanning electron microscopic examination.

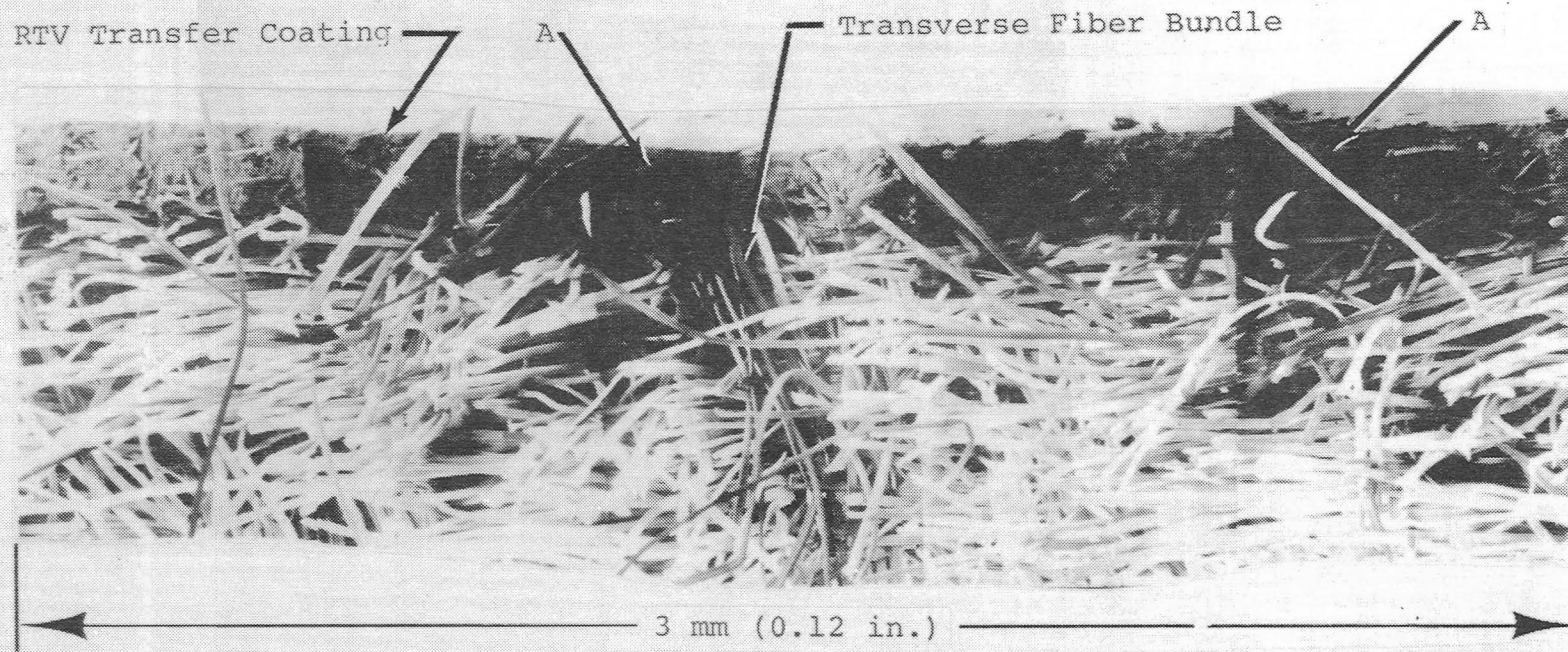




(a) Uncoated SIP face, 13X.



(b) Dimple in SIP face, 425X.



(c) Cross section of SIP showing fiber/transfer coat interface.

Figure 4.- Photomicrographs of Strain Isolation Pad (SIP).



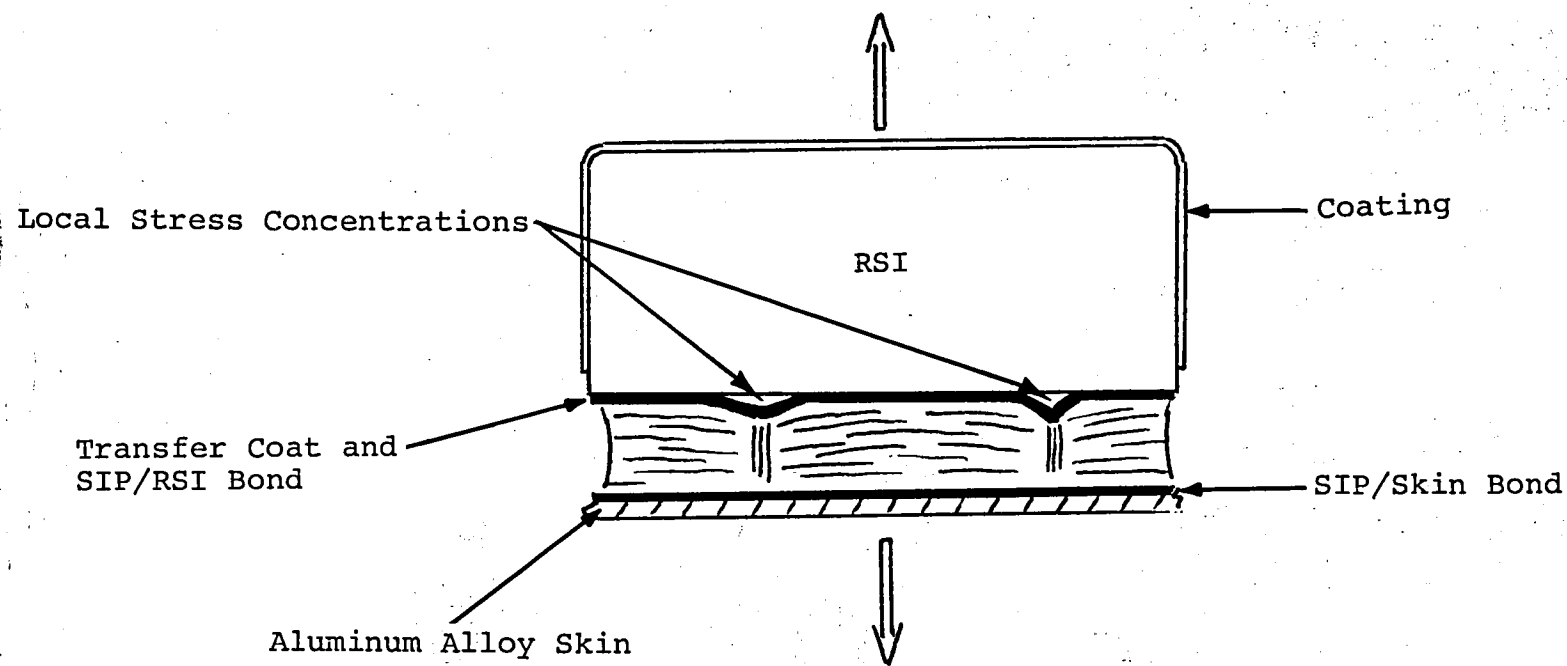
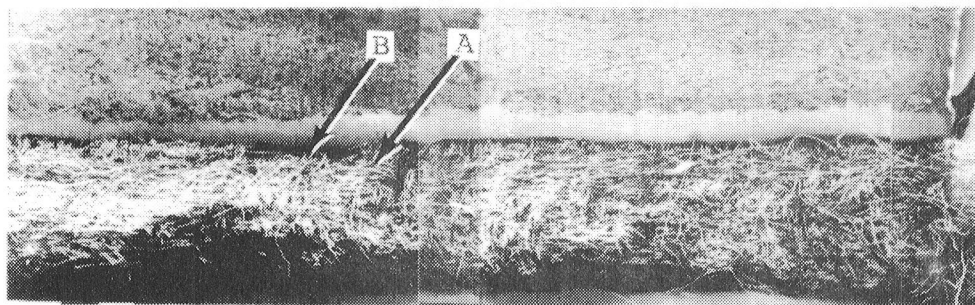
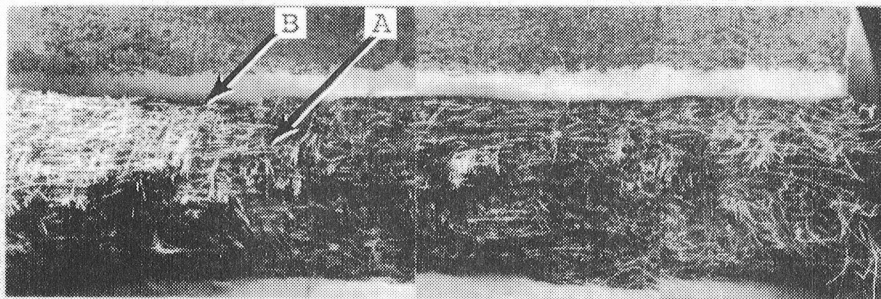


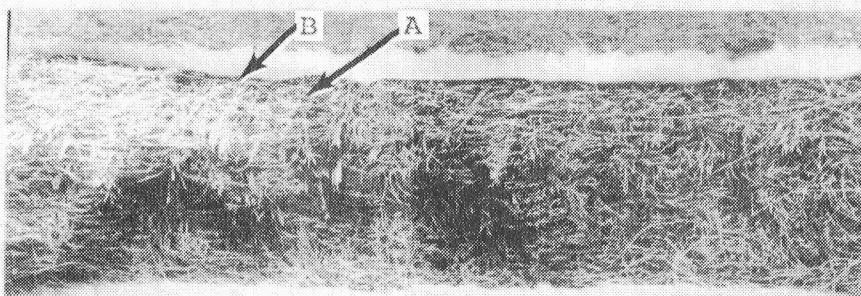
Figure 5.- RSI/SIP bond line immediately prior to failure under transverse tension load.



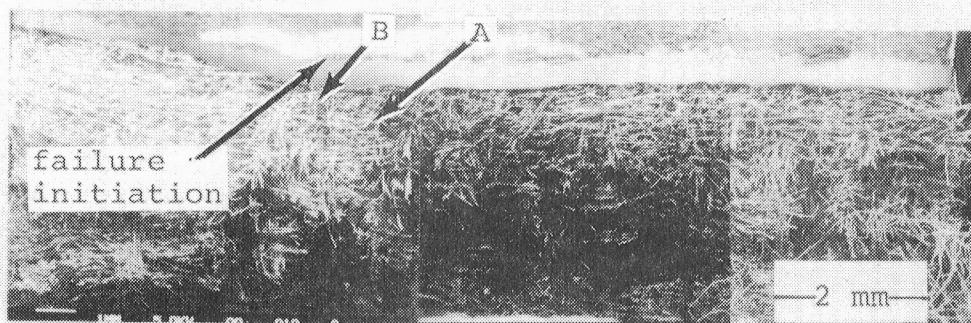
No load



$\approx 13.8$  kPa  
(2 psi)

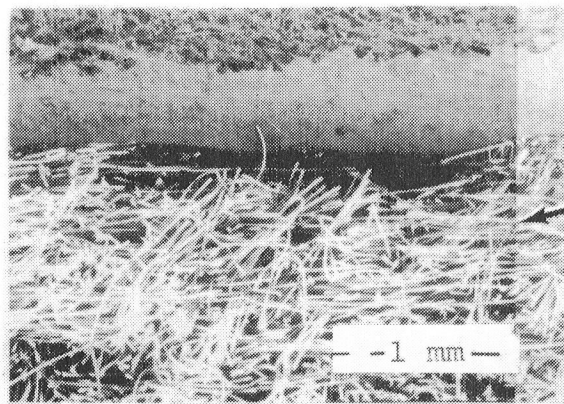


$\approx 43$  kPa  
(7 psi)

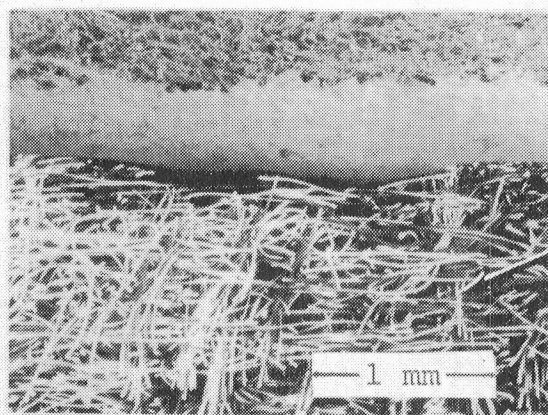


$\approx 110$  kPa  
(16 psi)

Figure 6(a).- SIP/RSI tension specimen cross section at different load conditions.



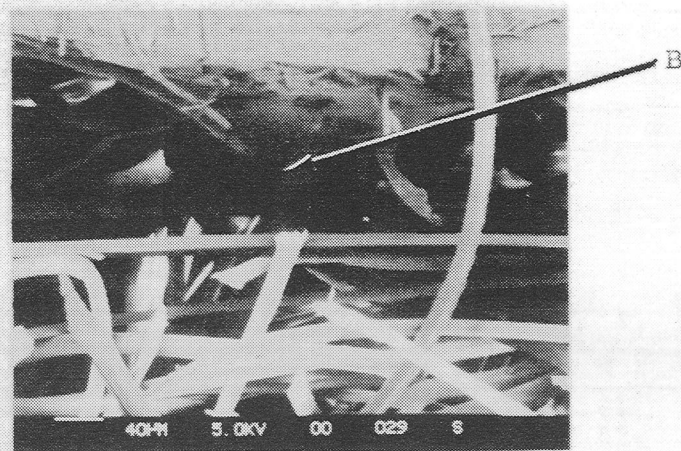
No load



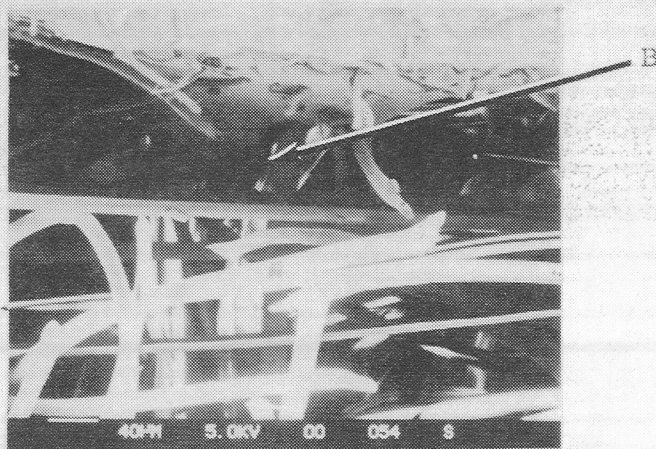
$\approx 13.8$  kPa (2 psi)

Figure 6(b).- Continued.

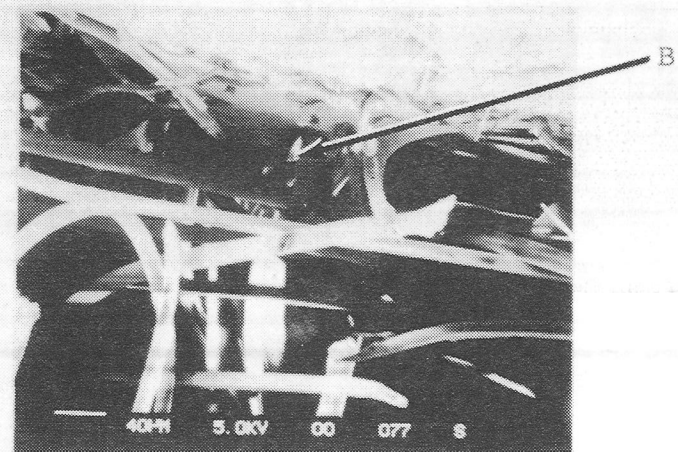




No load



$\approx 48$  kPa (7 psi)



$\approx 110$  kPa (16 psi)

Figure 6(c).- Concluded.

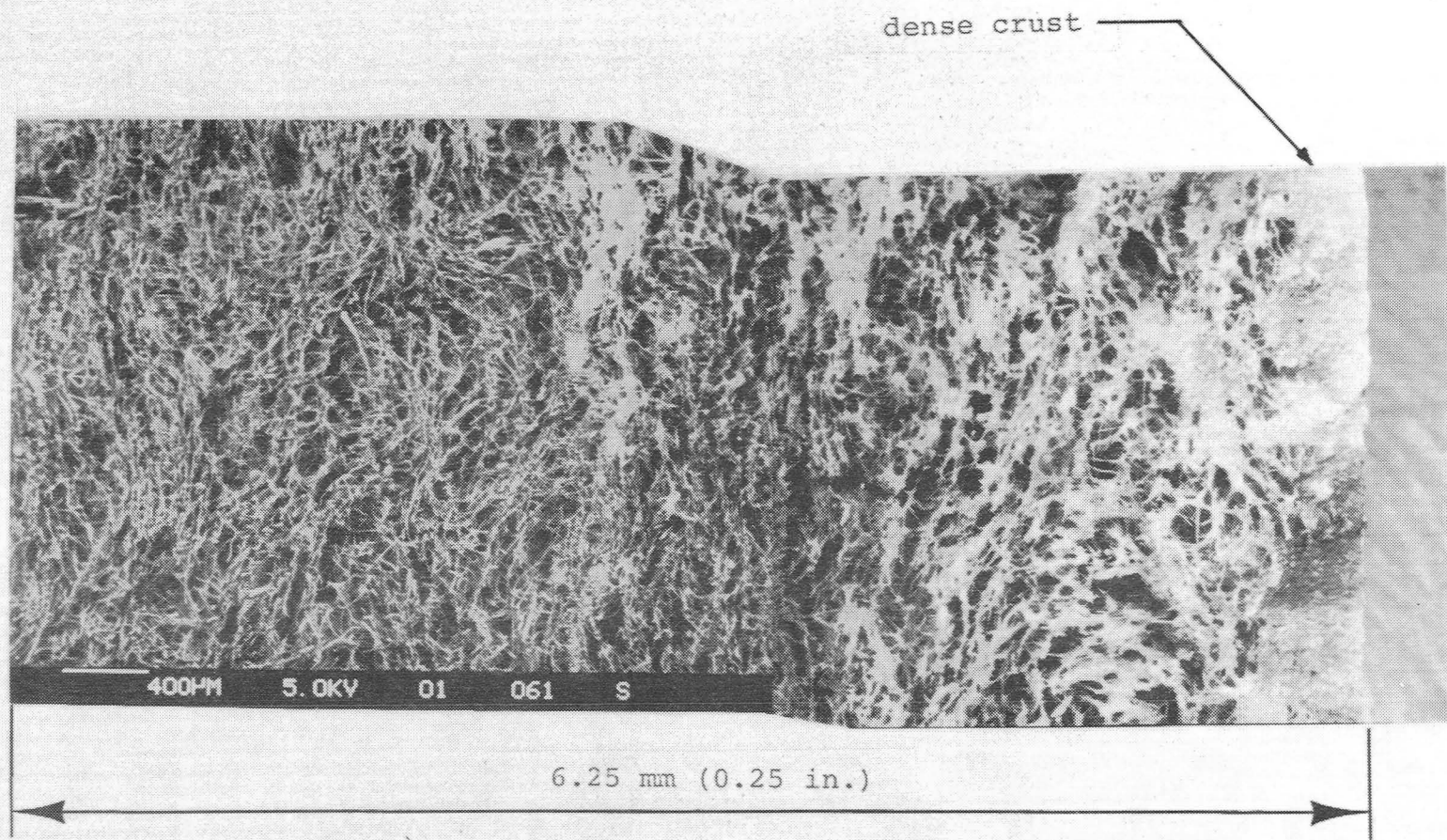
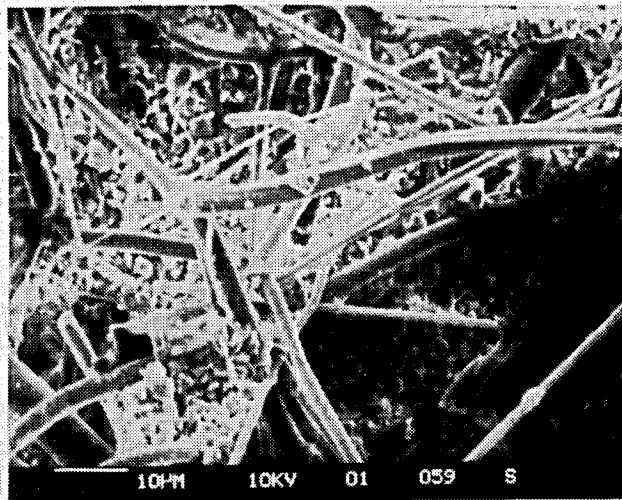
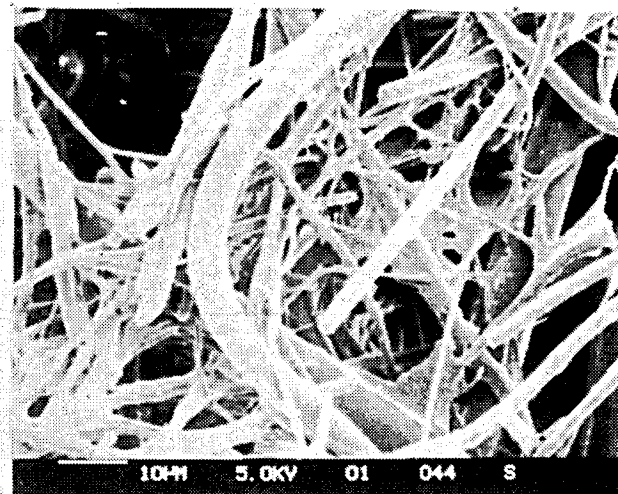


Figure 7.- Densified LI-900 HRSI cross section.

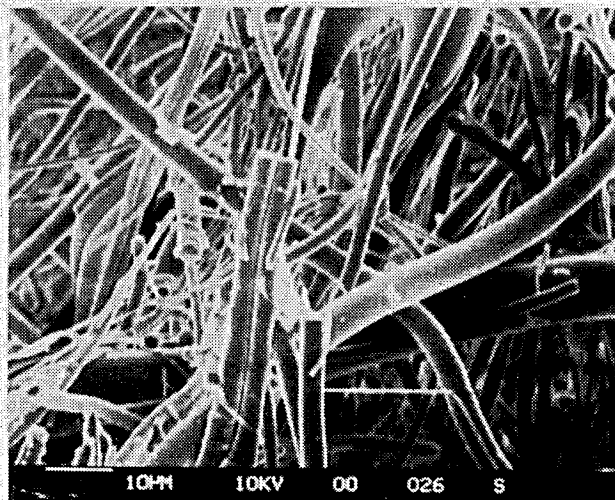




(a) Densified HRSI 2mm (0.08 in.) depth from bonding surface, 1350X.



(b) Densified HRSI 20mm (0.8 in.) depth from bonding surface, 1300X.



(c) Undensified HRSI, 1200X.

Figure 8.- Densified LI-900 HRSI compared with untreated HRSI.

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